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IS 356 (1991): Ester gum [CHD 20: Paints, Varnishes and Related Products]



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भारतीय मानक

ईस्टर गोंद (गम) — विशिष्ट

(दूसरा पुनरीक्षण)

Indian Standard

ESTER GUM — SPECIFICATION

(*Second Revision*)

UDC 665'944'2

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 2

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1952 largely based on the interim co-ordinated draft produced with the assistance of all concerned by the Co-ordinating Subcommittee of the No. 5 Standing Committee on Specifications for Paints and Allied Stores of the General Headquarters (now Army Headquarters) India. It was first revised in 1969 in order to align completely with IND/ME 178 'Resin ester gum lump size 100, for use in the manufacture of waterproofing composition RD1154' issued by the Department of Standardization, Ministry of Defence, Government of India. In the first revision, requirements for colour, acid value, ash content and softening point were modified. Additional requirements like gel time and residue on sieve considered essential by Defence users as also requirements like compatibility with drying oils and driers had been included.

The Committee responsible for the preparation of this standard decided to revise the standard so that the ester gum may also be used in nitrocellulose. In this revision, the requirement for compatibility with nitrocellulose has been added and the sampling procedure, acid value for glycerol ester have been modified. Gel flow in millimetres in 4 minutes has been substituted for gel time.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ESTER GUM — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for ester gum used in paints, varnishes, nitrocellulose, printing inks, certain waterproofing compounds, etc.

2 REFERENCES

The Indian Standards listed below are the necessary adjuncts to this standard.

IS No.	Title
354 (Part 1) : 1987	Methods of sampling and test for resins for paints : Part 1 General test methods (<i>second revision</i>)
460 (Part 2) : 1985	Specification for test sieves : Part 2 Perforated plate test sieves (<i>third revision</i>)
1070 : 1977	Water for general laboratory use (<i>second revision</i>)
1303 : 1983	Glossary of terms relating to paints (<i>second revision</i>)
1745 : 1978	Specification for petroleum hydrocarbon solvents (<i>second revision</i>)
6667 : 1972	Glossary of terms used in synthetic resin industry

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1303 : 1983 and IS 6667 : 1972 shall apply.

4 REQUIREMENTS

4.1 Description

The material shall consist wholly of rosin esterified with glycerol or other polyhydric alcohols. It shall be transparent and free from grit and visible foreign matter.

4.2 Solubility in Petroleum Hydrocarbon Solvents

The material shall be completely soluble in warm 145/205 low aromatic petroleum hydrocarbon solvent conforming to IS 1745 : 1978 and the solution shall be clear and free from turbidity at 30°C.

4.3 Colour

The colour of the material shall not be darker than a freshly prepared solution of 0.164 g of potassium bichromate in 100 ml of concentrated sulphuric acid when tested as prescribed in Annex A.

4.4 Compatibility with Linseed Oil

The material shall pass the test as prescribed in Annex B.

4.5 Compatibility with Nitrocellulose

The material shall pass the test as prescribed in Annex C.

4.6 The material shall also comply with the requirements prescribed in Table 1.

4.7 Additional Requirements for Defence User

4.7.1 Gel Flow in Millimetres in 4 Minutes

The gel prepared and tested as prescribed in Annex D shall be clear, homogeneous, sticky and shall flow through a distance of not more than 50 millimetres in 4 minutes.

4.7.2 Residue on Sieve

The material when screened through 150-micron IS Sieve [*see* IS 460 (Part 2) : 1985] shall leave no residue on the sieve.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in sound, clean and dry gunny bags or suitable packages as may be agreed to between the manufacturer and the purchaser.

5.2 Marking

Each package shall be marked with the following:

- a) Name of the material,
- b) Indication of the source of manufacture,
- c) Mass of the material,
- d) Batch number, and
- e) Month and year of manufacture.

6 SAMPLING

6.1 Representative samples of the product shall be drawn as prescribed in Annex E.

7 QUALITY OF REAGENTS

7.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Ester Gum
(Clause 4.6)

Sl No.	Characteristic	Requirement	Method of Test [Ref to Cl No. in IS 354 (Part 1) : 1987]
(1)	(2)	(3)	(4)
i) Softening point :			
a) Glycerol ester		90° to 100°C	10
b) Polyhydric alcohol ester		105° to 115°C	
ii) Volatile matter at 105±2°C, percent by mass, <i>Max</i>		0.50	12
iii) Matter insoluble in toluene, percent by mass, <i>Max</i>		0.20	13
iv) Ash, percent by mass, <i>Max</i>		0.20	14
v) Acid value, <i>Max</i> :			
a) Glycerol ester		11	15
b) Polyhydric alcohol ester		15	

ANNEX A
(Clause 4.3)

DETERMINATION OF COLOUR

A-0 GENERAL

A-0.1 Outline of the Method

The colour of the material made into a 50 percent solution (w/v) in suitable solvent is compared against that of freshly prepared solution of 0.164 g of potassium bichromate in 100 ml of concentrated sulphuric acid.

A-1 REAGENTS

A-1.1 Petroleum Hydrocarbon Solvent

Conforming to IS 1745 : 1978.

A-1.2 Potassium Bichromate Solution

Dissolve 0.164 g of potassium bichromate crystals in 100 ml of concentrated sulphuric acid.

A-2 PROCEDURE

A-2.1 Prepare a 50 percent (w/v) solution of the material in petroleum hydrocarbon solvent and transfer 10 ml of this solution to a thin-walled glass test-tube Nessler cylinder by means of a pipette. Take an exactly equal volume of potassium bichromate solution (A-1.2) in another test-tube, similar in every respect, for matching. Compare the colour of the two solutions holding the test tubes against light with a piece of moistened filter paper or Nessler cylinder glass interposed in between the light source and the test tubes.

NOTE — It is preferable to use a standard type of light source and viewing cabinet to cut off extraneous light.

A-2.1.1 The material shall be deemed to have passed the test if the colour of the solution made from the material is not darker than the potassium bichromate solution.

ANNEX B

(Clause 4.4)

TEST FOR COMPATIBILITY WITH LINSEED OILS

B-0 GENERAL

B-0.1 Outline of the Method

The material is cooked at specified temperature with linseed oil, boiled. The cooked material is allowed to form a bead on a glass plate and examined for haziness. To test the compatibility with driers, the cooked material is suitably thinned with petroleum hydrocarbon solvents, driers incorporated, and the resultant varnish examined for homogeneity at room temperature.

B-1 REAGENTS

B-1.1 Linseed Oil

B-1.2 Driers

B-1.3 Petroleum Hydrocarbon Solvents

Conforming to IS 1745 : 1978.

B-2 PROCEDURE

B-2.1 Heat one part of the material with one part of boiled linseed oil at 200°C with constant stirring and maintain at this temperature for about 30 minutes. Allow a little of the cooked material to form a bead on a glass plate cooling it to room temperature. Examine the bead when cold for haziness. The material shall be deemed to be compatible with the oil if the bead does not show any haziness.

B-2.2 Thin the material cooked as in B.2.1 with petroleum hydrocarbon solvents to give 50 percent non-volatiles at 140°C. Cool the material to 60° to 70°C and incorporate the drier in the proportion of one part of the drier to about 30 parts of the finished varnish. Cool the varnish to room temperature and apply a coat of the varnish on a clean glass plate by brushing. The material shall be taken to have passed the test if the varnish is clear and homogeneous and forms a clear and uniform film on the glass plate.

ANNEX C

(Clause 4.5)

TEST FOR COMPATIBILITY WITH NITROCELLULOSE

C-0 GENERAL

C-0.1 Outline of the Method

The material is dissolved in toluene and nitrocellulose solution is added to it. The resultant mixture is examined for transparency/clarity, both in the liquid condition, as well as after application in the form of a thin film on a glass panel and allowing it to dry.

C-1 REAGENTS

C-1.1 Toluene

C-1.2 *n*-Butanol

C-1.3 *n*-Butyl Acetate

C-1.4 Nitrocellulose

C-2 PROCEDURE

C-2.1 Prepare a solution of the material by dissolving in toluene by shaking in a closed tin container, to give a solution with a non-volatile matter content of 50 ± 1 percent (m/m.).

C-2.2 Prepare a solution of nitrocellulose by adding the following into a vessel and stirring with spatula:

Toluene	28.2 percent
Nitrocellulose	18.6 percent
<i>n</i> -Butanol	17.0 percent
<i>n</i> -Butyl Acetate	36.2 percent

Close the lid of the vessel tightly to prevent any loss of solvent and allow the nitrocellulose to completely dissolve, by keeping the solution for a minimum of 6 hours. After this, mix the solution with spatula to make it homogeneous. The non-volatile content of this solution shall be 13 ± 0.5 percent (m/m.).

C-2.3 Prepare a blend of 30 parts by weight of ester gum solution and seventy parts by weight of nitrocellulose solution, prepared as in C-2.1 and C-2.2, respectively, in a glass vessel. Examine the solution for visual clarity and freedom from haziness. The blend shall be clear and free from haziness.

C-2.4 Pour on a thoroughly cleaned glass panel of 150 mm × 100 mm, black tinted or with a black paint backing and allow to dry at ambient temperature for 20 minutes. Apply one more coat by pouring and allow to dry at ambient temperature for 4 hours. The coating shall not show any haziness even when viewed at a glazing angle.

ANNEX D
(Clause 4.7.1)

DETERMINATION OF GEL FLOW IN MILLIMETRES IN 4 MINUTES

D-0 GENERAL

D-0.1 Outline of the Method

The material is heated with a lubricating oil and aluminium stearate, and filled to a specified mark in a test-tube. This test-tube is then conditioned in an oven maintained at 60°C. Subsequently the test-tube is laid horizontally in the oven for exactly 4 minutes and the distance of flow of gel from original mark is noted.

D-1 REAGENTS

D-1.1 Spindle Oil

D-1.2 Aluminium Stearate

D-2 PROCEDURE

D-2.1 Heat 74.0 g of the material with 135 g of spindle oil in an aluminium beaker, about 75 mm internal diameter and 100 mm high, to 200°C in 30 minutes with constant stirring. Maintain the

material at this temperature for 30 minutes. Then cool it below 40°C and add 20 g of aluminium stearate and stir until a homogeneous mixture free from lumps is obtained. Heat the mixture carefully with constant stirring to 155° to 165°C for about 30 minutes and maintain at that temperature for another 30 minutes.

D-2.1.1 Fill a test-tube, 150 mm long and 25 mm internal diameter, with the hot gel to a depth of 50 mm and mark the tube outside at the surface of the gel. Allow the tube to cool to room temperature and then let it stand vertically for 3 hours in close proximity to a thermometer in an oven maintained at 60°C. Then lay the test-tube horizontally on a piece of wood in close proximity to the thermometer, avoiding appreciable changes in temperature and leave in this position, with the oven closed, for 4 minutes.

D-2.1.2 Measure the distance of flow of gel from the original mark on the tube.

ANNEX E
(Clause 6.1)

SAMPLING OF ESTER GUM

E-1 SAMPLING

E-1.1 Scale of Sampling

E-1.1.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

E-1.1.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers *n* to be selected from a lot *N* shall be in accordance with Table 2. The containers shall be selected at random.

E-1.2 Preparation of Test Samples

E-1.2.1 From each of the containers selected according to E-1.1.2, a small representative portion of the material shall be drawn with the help of the sampling scoop by inserting it into the material at different portions.

E-1.2.2 Out of the material drawn from individual containers, small but equal quantities of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determination for all the characteristics. If the quantities of material mixed from different portions are quite large, the process of coning and quartering may be adopted to obtain the requisite quantity.

NOTE --- On a clean impervious surface, heap the sample into a cone by depositing small representative quantities one on the top of the other, ensuring that the apex always remains in the same vertical line. Break down the cone, mix and re-form the cone twice in the same manner. Flatten the third cone by pressing with a metal or glass sheet so that a mass of uniform thickness and diameter is obtained, then quarter it along two diameters. Discard the diagonally opposite quarters and heap remaining two quarters into a cone. Repeat this process until a sufficient quantity is left to provide a sample.

E-1.3 Number of Tests

Tests for determination of all characteristics shall be conducted on the composite sample.

E-1.4 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification, if all the test results obtained after testing according to E-1.3 satisfy the relevant requirements of the specification.

Table 2 Scale of Sampling for Ester Gum

Lot Size	Number of Containers to be Selected
<i>N</i>	<i>n</i>
(1)	(2)
Up to 50	2
51 to 150	3
151 to 300	4
301 to 500	5
501 and above	6

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